Comparison of Vickers hardness between dental ceramics and hybrid materials

A THESIS

SUBMITTED TO THE FACULTY OF THE UNIVERSITY

OF MINNESOTA

BY

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IN PARTIAL FULFILLMENT OF THE REQUIREMENTS

FOR THE DEGREE OF

MASTER OF SCIENCE

ADVISOR:

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MAY 2024

Acknowledgments

I extend my heartfelt gratitude to the members of my thesis committee, Drs. Alvin Wee, Fengyuan Zheng and Arpit Nirkhiwale, for their invaluable contributions and dedicated efforts in reviewing and evaluating my work. Their insightful feedback, constructive criticism, and unwavering support have been instrumental in shaping the outcome of this thesis. I am sincerely thankful for their time, expertise, and commitment to excellence throughout this academic endeavor.

Abstract

The study aims to compare Vickers hardness of five definitive crown materials in a controlled laboratory setting. *Materials & Methods:* Five specimens (15 x 12 x 1.5 mm) were obtained from each tested material: zirconia (Lava Ultimate), lithium disilicate (IPS e.max CAD), lithium silicate reinforced with zirconia (Celtra Duo CAD), dual polymer hybrid ceramic (Vita Enamic) and resin ceramic hybrid (VarseoSmile Crown Plus). Vickers indenter (Wilson VH3100, Buehler) was used to create 6 pyramid-shaped indentations on each specimen and surface hardness was then measured. *Results:* A statistically significant difference in Vickers Hardness was found between all tested materials. Zirconia has the highest Vickers hardness while VarseoSmile BEGO has the lowest. Celtra Duo has a higher mean microhardness than Emax, yet still lower than Zirconia. Vita Enamic had lower microhardness than both Emax and Zirconia. VarseoSmile BEGO has lower microhardness than both Emax and Zirconia. *Conclusion:* Vickers Hardness for the tested materials can be classified as follows: Zirconia > Celtra Duo > Emax > Vita Enamic > Varseosmile BEGO.

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Introduction

Background

Material selection for dental restorations has a direct impact on success. This depends on multiple characteristics of the material that include mechanical properties, surface texture, translucency, and color stability among others. Until recently, the most commonly used esthetic restorative material was porcelain fused to metal. Many clinicians remain more comfortable using porcelain fused to metal because of its excellent and well-documented mechanical properties in the literature(1). More recently, there has been an increase in esthetic awareness amongst patients presenting for treatment. In this age and time, many patients associate success with good looks, which includes the teeth and smile. Multiple studies have described this trend, and some authors have concluded that people may not desire youthfulness as much as they desire youthful traits that include energy, intellect and, of course, a pleasing physical appearance(2). This has increased the demand for not only functional dentistry, but highly esthetic functional dentistry. Because of this shift, metal-free ceramic restorations have become increasingly popular, particularly in the anterior zone.

Literature Review

Dental Ceramics

Recent improvements in ceramic materials have given them an edge over porcelain fused to metal restorations. All-ceramic materials provide optimal esthetic outcomes due to their favorable optical properties, life-like luster, biocompatibility, chromatic stability and good mechanical properties(3). Today, the number of all-ceramic materials available for clinicians has increased substantially. This, in turn, has made it necessary to bring forward new classification systems that improve on the traditional system for classifying dental ceramics. According to recent classification systems (figure 1) (1,4), all-ceramic materials can be categorized into three main groups: (1) glass-matrix ceramics, (2) polycrystalline ceramics, and (3) resin-matrix ceramics.

Glass-matrix ceramics, as the name indicates, are inorganic ceramics that contain a glass phase or matrix. They are divided into three subclasses: feldspathic, synthetic, and glass-infiltrated ceramics. They differ from polycrystalline ceramics that lack a glass matrix and are composed of a crystalline phase. Zirconia is the most common polycrystalline ceramic, other subgroups include: alumina, zirconia-toughened alumina, and alumina-toughened zirconia. Resin-matrix ceramics are composed of an organic matrix highly filled with ceramic particles. They are split into various subgroups based on their composition (3,4).

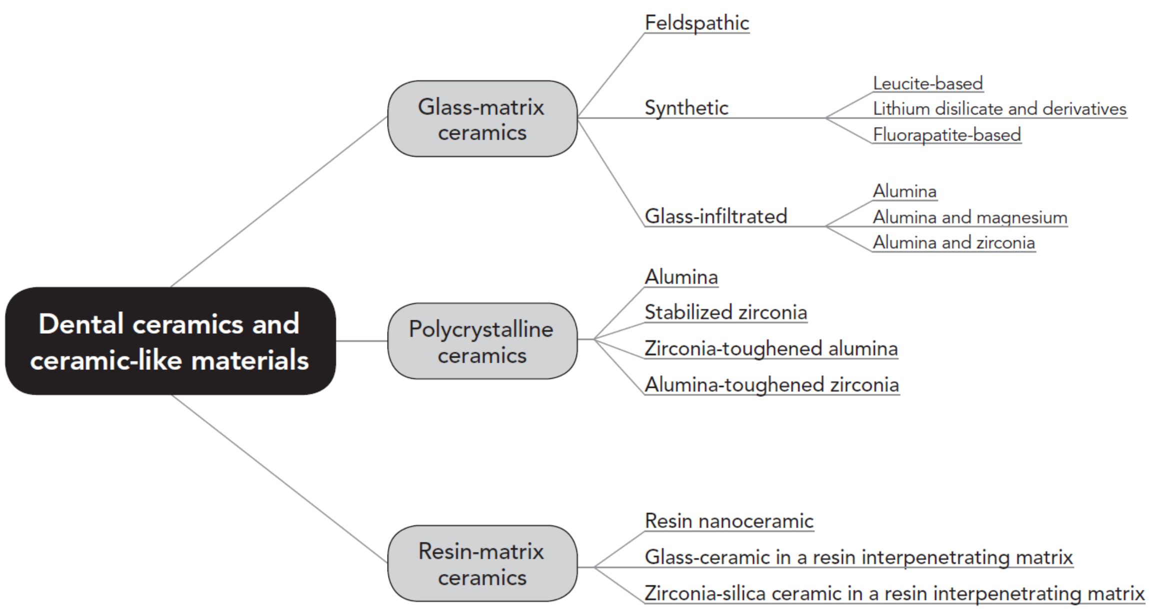

Figure 1: All-ceramic and ceramic-like materials as classified by Gracis et al. (2015) (4)

Lithium Disilicate

Lithium disilicate (LS_2) stands out as one of the most recognized and extensively utilized glass ceramic materials. Its composition includes quartz, lithium dioxide, phosphor oxide, alumina, potassium oxide, and various other constituents. The production of lithium disilicate involves a continuous manufacturing process based on glass technology that is optimized to systematically avert the occurrence of defects, notably pores. The microstructure of pressable lithium disilicate is characterized by the presence of around 70% needle-like crystals of lithium disilicate, which are intricately embedded within a glassy matrix. These crystals measure approximately 3-6 x 0.8 µm. The color of the lithium disilicate is gained by dispersing staining ions into the glassy matrix Variations in translucency levels are achieved by modifying the size and arrangement of crystals within the glassy matrix. Color-controlling ions are uniformly dispersed throughout the single-phase material, effectively eliminating any imperfections related to color pigmentation in the microstructure. Pressable lithium disilicate has a flexural strength that ranges from 450-500 MPa (5,6).

Milled lithium disilicate is similar in structure. The millable lithium disilicate crystals are formed by casting transparent glass ingots that contain lithium orthosilicate. Partial crystallization follows this process and results in the formation of lithium metasilicate crystals, which are now embedded in a glassy phase. These intermediate phase crystals give the CAD/CAM blocks their "blue" color, but also interfere with the material's processing characteristics, machinability, and good edge stability. This machinability is, in part, due to their size which ranges between 0.2-1 µm. After milling, these restorations undergo a heating cycle (840°-850 °C for 10 min) that turns the metasilicate crystals into lithium disilicate crystals ($Li_2Si_2O_5$). The formation of these crystals gives the restoration the tooth-shade, translucency as well as its flexural strength of approximately 360 MPa (5,7,8).

Zirconia

Pure zirconia is a metal oxide found in nature in three different crystalline configurations. The crystalline configurations depend on the temperature. The monoclinic configuration, which is stable up to 1,170°C, converts to the tetragonal form until 2,370°C, and finally to the cubic form when the temperature exceeds 2,370°C (9). The transformation from the tetragonal to the monoclinic configuration results in a volume increase, which can close cracks that form in the material. Due to its instability, pure zirconia cannot be utilized for dental restorations. This zirconia is stabilized with multiple oxides, namely yttrium. Other oxides used include magnesium, calcium and cerium (9). Dental zirconias are all commonly of the tetragonal zirconia polycrystal (TZP) type. Conventional dental zirconia contains a 3 mol% of yttrium-oxide and is known as 3Y-TZP zirconia (8,10). Under the energy resulting from different forces (mechanical, thermal or both), the atomic bonds of the zirconia structure break and result in a change from tetragonal to monoclinic crystals. This process is known as phase transformation toughening (11). The stabilized tetragonal zirconia (3Y-TZP) used in clinical practice positively utilizes this transformation toughening to produce restorations that withstand incredible forces of over 1,000 MPa (12).

One of the disadvantages of zirconia as ceramic for dental restoration is the increased opacity and its lack of translucency. This is why zirconia is considered esthetically inferior to glass-ceramics such as lithium disilicate. However, the increased opacity makes zirconia a suitable choice to mask dark substrates such as dark stump shades and cast cores. In an attempt to improve the esthetic properties of zirconia, manufacturers attempted to increase translucency of the material by adding an increased yttrium content of 5 mol% to cubic zirconia and introduced 5Y-TZP zirconia to the market. Although the new 5Y-TZP zirconia product has significantly improved esthetic and optical properties, the cubic form of zirconia utilized in this product does not undergo phase transformation toughening and therefore did not have the same strength and fracture resistance that made 3Y-TZP such a popular restorative material. The resultant 5Y-TZP zirconia has a flexural strength ranging between 500-900 MPa (8,13). A study by Kwon et al. (6) showed that 5Y-TZP zirconia has a flexural strength that ranks between 3Y-TZP and lithium disilicate, but studies on bonded lithium disilicate crowns showed that adhesively bonding to dentin gives the lithium disilicate support that allows it to withstand loads that are significantly higher than those of 5Y-TZP zirconia (14,15).

In an attempt to combine the strength of zirconia with the esthetics of feldspathic porcelain, bilayered zirconia, or porcelain fused to zirconia (PFZ) was introduced. The concept was similar to that of porcelain fused to metal, whereby a thin zirconia coping is veneered with a layer of feldspathic porcelain. Although the results were esthetically superior to monolithic zirconia, fracture or “chipping” of the veneering porcelain is a common complication. Hamza and Sherif’s recent publication showed that monolithic lithium disilicate is more resistant to fracture than bilayered zirconia (16).

Zirconia-Reinforced Lithium Silicate

The information reviewed on the commonly used all-ceramic materials shows that the field of prosthodontics and restorative dentistry would benefit greatly from a ceramic that combines the esthetic and optical properties of lithium disilicate with the high flexural strength of monolithic zirconia. In an attempt to fill this need Vita introduced Vita Suprinity PC, a zirconia-reinforced lithium silicate (ZLS) CAD/CAM block that can be used for the fabrication of anterior and posterior full and partial coverage crowns. This new glass ceramic has been reinforced with 10% zirconium (by weight), and the manufacturer claimed that the material combines the optical properties of glass ceramics in addition to the flexural strength and characteristics of zirconia (17). Shortly after, Dentsply Sirona introduced Celtra Duo (figure 2), their own brand of zirconia-reinforced lithium disilicate blocks. Dentsply Sirona claimed that the increased glass content of the ZLS ceramic improves translucency and esthetics, where the dissolved zirconia (10% by volume) reinforces the ultrafine microstructure of the glass matrix without the clouding that is typical of zirconia ceramics. Dentsply Sirona later introduced ingots of the same material that could be used with traditional pressable ceramic technology under the name Celtra Press (17,18).

Its composition includes lithium metasilicate (Li_2SiO_3) and lithium orthophosphate crystals (Li_3PO_4), along with around 10% zirconium dioxide (ZrO_2) for reinforcement. Through a final process of crystallization, a fine-grained microstructure comprising Li_2O-ZrO_2-SiO_2 is formed.

ZLS can be marketed in pre-crystallized or crystallized forms. The partially crystallized form is often preferred for CAD/CAM manufacturing, as it facilitates machining. Subsequent treatment to complete crystallization is performed to achieve the desired final color and optimal mechanical properties (19). This process ensures that the material meets both aesthetic and functional requirements for dental restorations.

As per the manufacturer's specifications, Celtra Duo has high flexural strength of 210 Mpa in the uncrystallized state and gets to 370 Mpa heat after crystallization.

Figure 2: Celtra Duo Blocks (20)

In contrast to zirconia restorations, the manufacturer's guidelines indicate that ZLS can be etched and bonded using adhesive systems (21). Studies have shown that the fracture resistance of monolithic CAD/CAM ceramic crowns, when adhesively cemented, is significantly greater than those cemented through conventional methods (22).

Elsaka & Elnaghy’s study comparing zirconia-reinforced lithium silicate blocks (Vita Suprinity PC, Vita) to lithium disilicate blocks (IPS e.max CAD, Ivoclar Vivadent) found that the ZLS blocks revealed better mechanical properties compared to the lithium disilicate blocks (18).

Kashkari et. al. compared the load-to-fracture values of monolithic zirconia, lithium disilicate and zirconia-reinforced lithium silicate crowns. Their results showed that zirconia had the highest load-to-fracture values, followed by lithium disilicate and finally the ZLS. They concluded that more research should be conducted before using ZLS for molars in patients with parafunctional habits (23).

Research by Bergamo et. al. on the thickness and survival mode of bonded zirconia-reinforced lithium silicate crowns showed that 0.5 mm thickness exhibited significant reduction in probability of survival when compared to crowns that were 1-1.5 mm in thickness (24). When compared with the findings of Sasse et. al. who conducted a similar study on bonded occlusal lithium disilicate restorations, the results were similar. Sasse found that 0.7-1 mm of thickness is suggested for bonded occlusal lithium disilicate restorations (25). Another study by Chen showed that minimal thickness lithiumle disilicate crowns (0.7 mm occlusal thickness) did not offer statistically reduced fracture resistance when compared to traditional thickness lithium disilicate crowns that had an occlusal thickness of 1.5 mm (26). The comparison between the results suggests that, within the limitations of the studies, both materials appear to be similar in strength.

Hybrid Ceramics = Resin-matrix ceramic

This category encompasses materials featuring an organic matrix extensively filled with ceramic particles. (> 50% by weight). These hybrid materials were developed by manufacturers to find materials that (4):

1. simulate more closely the flexural strength, modulus of elasticity and hardness of natural tooth structure as compared to traditional materials (27,28)
   Flexural strength of PICN is 150 MPa whereas that of nano-ceramics is 200 MPa (27,29–31)
2. Absorb chewing forces more than glass ceramics (29)
3. develop a material easier to mill and adjust
4. Enable the ease of repair or modification using composite resin.

Resin-matrix ceramic composition differs largely, which affects the material's resistance to mechanical and chemical degradation.
These hybrid materials can be categorized into several subgroups depending on their inorganic composition.

a- *Resin nanoceramic* (eg, Lava Ultimate, 3M ESPE)

It is composed of a highly cured resin matrix strengthened with around 80% by weight nanoceramic particles, predominantly silica, zirconia and their bound aggregates (< 20 nm in diameter).
These nanoceramics minimize the gaps between filler particles, allowing for a high content of nanoceramic material (32).
Lava Ultimate exhibits enhanced translucency to VITA Enamic and glass ceramics, owing to its finer filler size. Consequently, it is suitable for applications such as inlays, onlays, and veneers. However, it is no longer recommended for full crowns due to issues related to debonding (33–36).

b- *Zirconia-silica ceramic in a resin interpenetrating matrix*

This subcategory exists with a variety of organic matrices and with different ceramic weight percentages. Examples include:

- Shofu Block HC (Shofu) and MZ100 Block
  - organic content: UDMA, TEGDMA
  - inorganic content: 60 % by weight of silica powder, zirconium silicate, micro-fumed silica
- Paradigm MZ-100 Blocks, 3M ESPE
  - organic content: bisGMA, TEGDMA, and a patented ternary initiator system.
  - inorganic content: 85 % by weight of very fine zirconia-silica ceramic particles.

c- *Glass ceramic in a resin interpenetrating matrix = Polymer-infiltrated ceramic network (PICN) material*

These materials are composed of a dual network: inorganic ceramic and organic polymer, and they combine positive characteristics of both all-ceramic and composite materials.

- **Vita Enamic**

It consists of a dual network structure: a partially sintered feldspathic network (86% by weight - 75% by volume) and a reinforcing polymer network (14% by weight - 25% by volume).

The ceramic network is mainly composed of Silica (58-63% SiO_2) and Alumina (20-23% Al_2O_3). The organic polymer network is formed by urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA) and mainly aims to reduce tendency to brittle fracture (4,37).

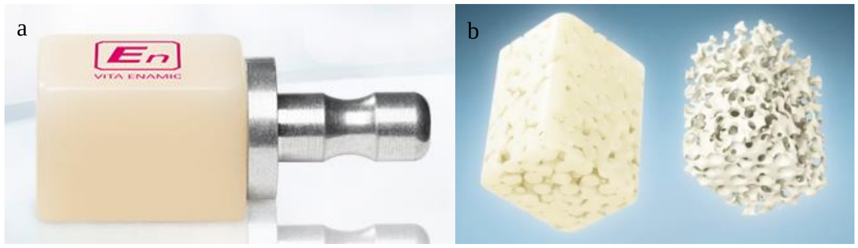

Figure 3: Vita Enamic block (figure 3a) with a unique dual ceramic-polymer network structure (figure 3b) (37).

This material presents with increased porosity which consequently increases flexural strength while reducing its hardness. Additionally, it is believed that the ductile polymer phase in the PICN material enhances strength and toughness through effective crack bridging (38).

VITA Enamic was noted to exhibit lower level of translucency compared to Lava Ultimate and glass-matrix ceramics, attributed to its relatively high concentration of Al_2O_3 (33,34).

- **<u>VaseoSmile Crown Plus (BEGO, Bremen, Germany)</u>**

The emergence of 3D printed permanent restorations utilizing a hybrid ceramic material composited of ceramic filled methacrylate-based resin has revolutionized the market, as all materials that have been available for 3D printing have been FDA approved as provisional or interim prostheses (39). According to the manufacturer, VarseoSmile Crown Plus (BEGO, Bremen, Germany) can be used for single crowns, inlays, onlays, and veneers (40).

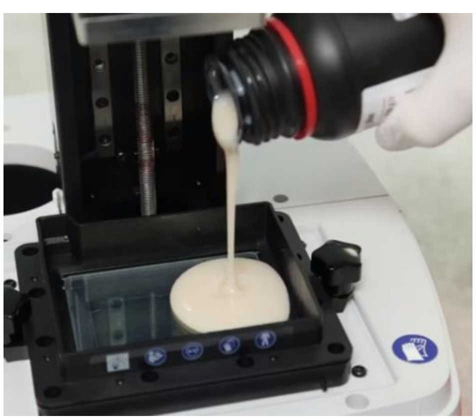

Figure 4: VarseoSmile Crown plus resin (41).

These hybrid resin ceramics have shown a flexural strength of 116-150 MPa with a breaking load of 1,936 N in chewing simulations.

Holmer et. al. (42) compared the shear bond strength of the VarseoSmile Temp material (BEGO, Bremen, Germany) using Variolink Esthetic and Fuji Cem 2 and compared different surface treatments. In this study, Variolink Esthetic had superior bond strength than Fuji Cem 2, although alumina pre-surface treatment improved the strength of Fuji Cem 2 with shear bond testing reporting 7,447 MPa and 4,921 MPa, respectively. However, limited data and studies on the flexural strength of these materials are available.

Mechanical testing

Mechanical testing of dental materials involves a variety of assessments to ensure that the materials used in restorative dentistry meet specific criteria for strength, durability, and other mechanical properties. Some common mechanical tests conducted on dental materials include hardness, tensile, compression, flexural, shear, fatigue, impact, wear and facture toughness testing.

These tests help assess the mechanical behavior of dental materials under conditions that simulate the oral environment. The results of these tests guide dental professionals in selecting appropriate materials for various dental applications, considering factors like strength, durability, and wear resistance.

Vickers Hardness

Vickers hardness is a measure of a material's hardness based on its resistance to indentation. It is named after its inventor, Smith and George E. Sandland, who developed the Vickers hardness test in 1924 (43). The Vickers hardness test is widely used in various industries, including materials science and engineering, to assess the hardness of metals, ceramics, and certain types of polymers.

Test method includes (44):

1. **Test specimen:**
    - Thickness: the test remains unaffected provided that the specimen is more than ten times thicker than the indentation depth. Generally, a thickness of at least 0.50 mm ensures that hardness measurements are not impacted by variations in thickness.
    - Surface: pecimens should exhibit a ground and polished surface with a roughness of less than 0.1 µm rms.
2. **Indentation:**
    - A square-based diamond pyramid with 136̊ face angles indenter is pressed into the material being tested under a specified force.

- The indentations should be separated by a minimum of four diagonal lengths between the centers of the indentations.
- If indentations result in cracking, the spacing must be expanded to a minimum of five times the length of the cracks.

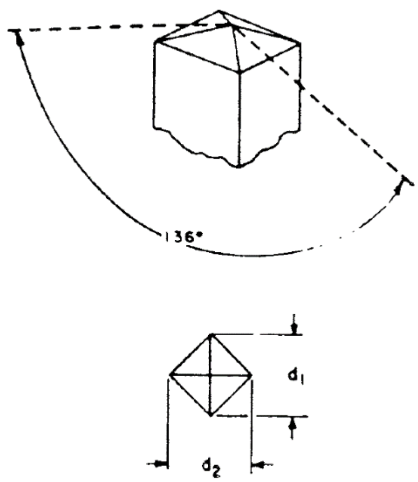

Figure 5: Vickers indenter: a square-based diamond pyramid with 136° face angles indenter.

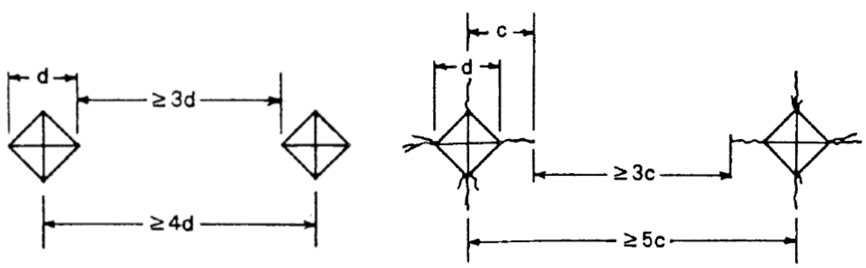

Figure 6: Spacing of Vickers Indentations: a minimum distance of 4 diagonal lengths (d) should be present between the centers of the indentations. In case of cracking, the minimum distance between centers increases to a minimum of five (d).

3. **Measurement:** The size of the resulting square-shaped indentation is measured diagonally with an optical microscope.

4. **Calculation:** The Vickers hardness number (HV) is determined by dividing the applied load over the surface of the indentation using the formula:

$$HV = \frac{2 \times \text{Force (in Newtons)}}{\text{Diagonal length of the indentation (in millimeters)}^2}$$

Vickers hardness can be expressed either in Gigapascals (GPa) or as Vickers hardness number. Higher Vickers hardness values indicate greater hardness and resistance to deformation. The Vickers hardness of ceramics generally diminishes as the indentation size or indentation force increases.

Hardness is a crucial property when evaluating dental restorative materials. Surface hardness, as a relative measure of a material's resistance to permanent surface indentation or penetration, plays a significant role in determining the abrasiveness of the material and its potential impact on natural dentition (29,45). The rationale behind measuring hardness in dental materials is to assess how these materials may affect the wear and integrity of natural teeth when subjected to chewing forces and other oral activities. Dental porcelains and ceramics have been observed in various wear test studies to exhibit higher levels of antagonist tooth wear compared to other restorative materials. This indicates that these harder materials may cause more substantial wear on the opposing natural teeth (46–48).

Understanding the hardness of dental materials is crucial for dental practitioners and researchers to select appropriate restorative materials based on the specific requirements of a patient. It allows them to consider the potential impact of a material's hardness on the longevity and health of the restored teeth as well as its influence on the wear characteristics of the natural dentition (49).

Study aim

The purpose of this study was to measure Vickers Hardness of several CAD/CAM materials, including lithium disilicate (e.max CAD), Zirconia (Lava Esthetic), lithium silicate/zirconia (Celtra Duo), a polymer infiltrated ceramic (Vita Enamic), and resin ceramic hybrid (VarseoSmile Crown Plus).

The null hypothesis is that there’s no difference in Vickers Hardness between all tested materials.

Materials & Methods

Study design

The comparison of mechanical properties of 5 definitive crown CAD/CAM materials was conducted. The tested materials are: zirconia (Lava Ultimate), lithium disilicate (IPS e.max CAD), lithium silicate reinforced with zirconia (Celtra Duo CAD), dual polymer hybrid ceramic (Vita Enamic) and resin ceramic hybrid (VarseoSmile Crown Plus). Their composition, as outlined in the literature, is presented in Table 1.

Specimen preparation

Five square-shaped specimens (15 x 12 x 1.5 mm thick) were obtained from each CAD-CAM material according to ISO 6872:2015 (50) and ASTM C1327-15(2019) (44).

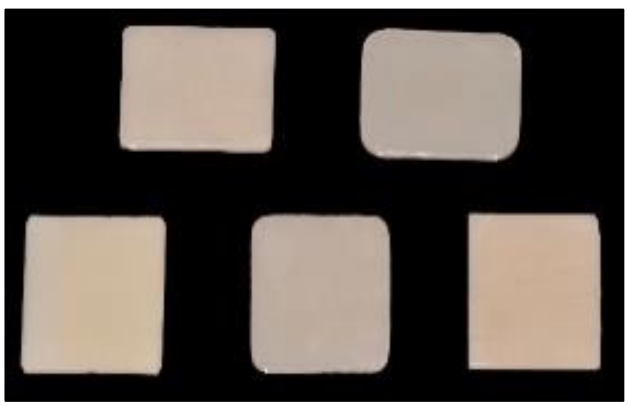

Figure 7: Specimens (15 x 12 x 1.5 mm) obtained from each CAD-CAM material.

The CAD/CAM blocks (LS_2 – ZLS – Vita enamic) were cut using low-speed water-cooled diamond saw (Isomet, Buehler, GmbH, Dusseldorf, Germany).

A three-dimensional digital design of the Zirconia and VarseoSmile specimens was created using MEDIT Link.

Zirconia specimens were then milled with DWX-52DCi 5-Axis dental milling machine (Roland DGA, USA) then sintered with Upcera GT1 Zirconia sintering furnace (UPCERA, China).

VarseoSmile Crown plus specimens were printed using Asiga Max (Asiga, Sydney, Australia) with automatically generated standard medium size supports with 75% density. Ethanol was used for post-processing. Final curing was completed in Form Cure (Formlabs, Somerville, MA, USA), 90 seconds on each side.

Celtra Duo and Emax specimens were then fully crystallized using Programat P500 furnace (Ivoclar-Vivadent, Schaan, Liehtenstein). All samples were then finished and polished in accordance with guidelines provided by the respective manufacturer (details summarized in table 1). The samples were then stored dry at room temperature.

Material	Classification	Manufacturer	Chemical content (wt%)	Specimen Preparation
IPS Emax CAD	Lithium Disilicate - Synthetic glass-matrix ceramic	Ivoclar Vivadent AG, Liechtenstein	58-80% SiO_2 11-19% Li_2O 0-13% K_2O 0-8% ZrO_2 0-5% Al_2O_3	- Cut using low-speed water-cooled diamond saw (Isomet, Buehler) - Polished via rubber and silicon polishers - Thermal treatment in Programat P510, Ivoclar
Lava™ Esthetic Zirconia Disc	Zirconia - Polycrystalline ceramic	3M, US	Zirconium dioxide Yttrium oxide (5 mol%).	- 3D digital design of the specimen was created using MEDIT Link - Milled with DWX-52DCi (Roland) dental milling machine - Sintered with UPCERA

Vita Enamic	Dual polymer hybrid ceramic - Resin-matrix ceramic	VITA Zahnfabrik, Germany	86% inorganic: *58-63%* SiO_2 *20-23%* Al_2O_3 *9-11%* Na_2O *4-6%* K_2O *0.1%* ZrO_2 14% organic: *Methacrylate polymer (UDMA, TEGDMA)*	- Cut using low-speed water-cooled diamond saw (Isomet, Buehler) - Polished via Vita Enamic polishing burs
Celtra Duo CAD	Lithium silicate reinforced with Zirconia	Dentsply Sirona, US	58% SiO_2 18.5% Li_2O 10.1% ZrO_2 1.9% Al_2O_3 1% Tb_4O_7 2% CeO_2 5% P_4O_{10}	- Cut using low-speed water-cooled diamond saw (Isomet, Buehler) - Polished via diamond polishing bodies - Thermal treatment in Programat P510, Ivoclar
VarseoSmile Crown Plus	Resin-matrix ceramic	BEGO, Bremen, Germany	Dental glass Methyl benzoyl formate Diphenyl (2,4,6-trimethylbenzoyl) Phosphine oxide 30-50% inorganic fillers (particle size 0.7 µm)	- 3D digital design of the specimen was created using MEDIT Link - Printed with Asiga Max printer - Final curing performed in Form Cure (Formlabs), 90 seconds on each side.

Table 1: Characteristics and specimen preparation of the five CAD-CAM tested materials.

Vickers Hardness

Vickers hardness was measured using a digital microhardness tester (Wilson VH3100, Buehler) using a 1 kgf (= 9.8 N) load for 20 s dwell time according to the ASTM C1327-03 standard (44).

Figure 8: Wilson VH3100 (Buehler) indenter

A pyramid-shaped diamond indenter with 136° face angles was used to create thirty indentations per material (six per specimen). The indentations were separated by a minimum of four diagonal lengths between the centers of the indentations.

Diagonal lengths of the Vickers indent were then measured by optical micrometer and Vickers Hardness (HV) was automatically calculated by dividing the force applied by the surface area of the indentation.

Statistical analysis

Statistical analysis was performed using linear mixed effect model that accounts for the correlation between six different measurements from every specimen.

The P-value was adjusted using Benjamini–Hochberg Method to account for multiple comparisons. The unadjusted p-value is smaller than the adjusted p-value.

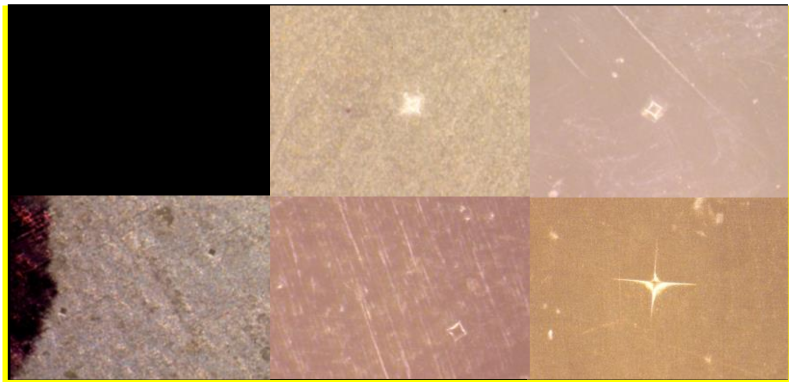

Figure 9: Vickers indentations on the five tested CAD-CAM materials.

Results

Mean Vickers hardness

Vickers hardness values for all tested specimens are listed in table 2 along with mean values of all materials.

Specimen	Indentation	Zirconia	Emax	Celtra Duo	Vita Enamic	BEGO
Specimen 1	1	905.4	481	556.4	204	21.9
	2	721.9	482.4	532.8	179.6	22.7
	3	860.6	427	530.5	188.4	30.5
	4	966.9	530.8	507.3	171.2	22.1
	5	870.4	515.9	560.3	152	22.2
	6	868.1	487.7	563.6	160.9	22.4
Specimen 2	1	824.5	420.1	550.2	195	21.7
	2	1201	481.6	549.7	191.8	18.7
	3	939.3	491.6	506.3	144.2	21.1
	4	946.5	509.1	527.8	171.9	21
	5	928.9	478.6	546.3	178.2	21.7
	6	877.1	504.3	540.1	172.6	22.5
Specimen 3	1	1061	465.6	564.3	171.1	21.8
	2	922.1	442.2	516.6	198.8	21
	3	993.1	448	822.3	164.2	20.3
	4	1013	421.3	522.9	196.8	21.2
	5	953.2	476.8	542.3	190	20.2
	6	979.1	466.7	553.7	176.9	21.3
Specimen 4	1	1053	473.7	537.3	185.7	20.7
	2	1051	472.4	529.2	158.8	20.1
	3	1137	497.7	536.3	209.1	20.1
	4	921.6	496.3	519.3	207.6	20.9
	5	897	480.8	532.7	168.1	21.1
	6	1017	521.8	515	167.6	20.2
Specimen 5	1	967.2	496.5	536.7	184.3	19.9
	2	936.4	485.4	540.2	190.7	20.6
	3	973.3	503.3	555.8	183.4	20.6
	4	1006	484.7	514.5	194.8	20.3
	5	866.2	520	526.2	156.3	23
	6	900.2	474	538.4	161.3	20.6
Mean (SD)		**951.9 (94.2)**	**481.2 (28.2)**	**545.8 (54.7)**	**179.2 (17.0)**	**21.4 (2.0)**

Table 2: Vickers hardness values

The mean (standard deviation) microhardness values of the tested materials are as follows:

- Zirconia (Lava Ultimate): *951.9 (94.2)*
- Lithium disilicate (IPS e.max CAD): *481.2 (28.2)*
- Lithium silicate reinforced with zirconia (Celtra Duo CAD): *545.8 (54.7)*
- Dual polymer hybrid ceramic (Vita Enamic): *179.2 (17.0)*
- Resin ceramic hybrid (VarseoSmile Crown Plus): *21.4 (2.0)*

The following bar plot displays the mean Vickers hardness for each material:

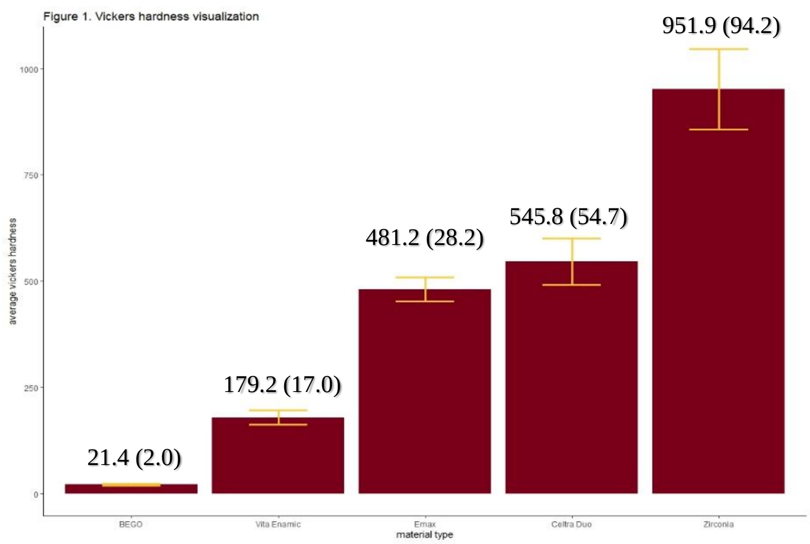

Figure 10: Bar plot displaying mean Vickers hardness for each CAD-CAM material.

Comparison between every pair of materials was performed using linear mixed effect. The results are shown in table 3. Linear mixed effect model showed statistically significant differences between materials for Vickers hardness ($p \leq 0.001$).

Zirconia had the highest Vickers hardness. Celtra Duo had higher mean microhardness than Emax, yet still lower than Zirconia. Vita Enamic had lower microhardness than Emax, Celtra Duo and Zirconia. VarseoSmile BEGO showed the lowest Vickers hardness among tested materials.

Table 3: Comparison between every pair of materials.

Material	Mean (SD)	Difference estimate[1]	95% CI	p-value[2]
Celtra Duo	545.8 (54.7)	-524.4	(-545.1,-503.8)	<0.001
BEGO	21.4 (2.0)			
Celtra Duo	545.8 (54.7)	-366.7	(-387.5,-345.8)	<0.001
Vita Enamic	179.2 (17.0)			
Celtra Duo	545.8 (54.7)	406.1	(352.9,459.3)	<0.001
Zirconia	951.9 (94.2)			
Celtra Duo	545.8 (54.7)	-64.6	(-89.7,-39.5)	0.001
Emax	481.2 (28.2)			
BEGO	21.4 (2.0)	157.8	(151.6,163.9)	<0.001
Vita Enamic	179.2 (17.0)			
BEGO	21.4 (2.0)	930.5	(881.5,979.5)	<0.001
Zirconia	951.9 (94.2)			
BEGO	21.4 (2.0)	459.8	(445.5,474.1)	<0.001
Emax	481.2 (28.2)			
Vita Enamic	179.2 (17.0)	772.8	(723.7,821.8)	<0.001
Zirconia	951.9 (94.2)			
Vita Enamic	179.2 (17.0)	302.1	(287.5,316.7)	<0.001
Emax	481.2 (28.2)			
Zirconia	951.9 (94.2)	- 470.7	(-521.7,-419.7)	<0.001
Emax	481.2 (28.2)			

1: The estimate difference is calculated by bottom row mean minus top row.

2: The p-value is adjusted using Benjamini–Hochberg Method to account for multiple comparison. The unadjusted p-value is smaller than the adjusted p-value. Since the adjusted pp-value is already significant, the unadjusted p-value wasn't reported.

Discussion

The present study was conducted to assess and compare Vickers hardness of five definitive crown materials: zirconia (Lava Ultimate), lithium disilicate (IPS e.max CAD), lithium silicate reinforced with zirconia (Celtra Duo CAD), dual polymer hybrid ceramic (Vita Enamic) and resin ceramic hybrid (VarseoSmile Crown Plus).

The results show significant differences and thus allow rejection of the null hypothesis.

Vickers Hardness for the tested materials can be classified as follows: Zirconia > Celtra Duo > Emax > Vita Enamic > VarseoSmile BEGO. Among all three test materials, only fired Celtra Duo surpassed Emax's hardness. Vita Enamic and Varseosmile showed significantly lower Vickers hardness than Emax.

These results are comparable to findings from several studies in the literature. Table 4 summarizes Vickers hardness values of different CAD-CAM definitive crown materials evaluated in scientific literature.

In a study comparing mechanical properties of Zirconia reinforced lithium disilicate ZLS (Vita Suprinity) to Emax, Elsaka et al. (2016) (51) confirmed that ZLS has statistically higher Vickers hardness than Lithium disilicate. Emax is thus considered a less abrasive material than Celtra Duo, creating less wear on the opposing dentition.

Lawson et al. (2016) (38) compared mechanical properties of Emax, fired and unfired Celtra Duo, Vita Enamic and three other composite materials. Celtra Duo and Emax were significantly harder than Vita Enamic with fired Celtra Duo being the hardest out of them.

Hardness of a material reflects its ease of milling: a harder material like Celtra Duo will require more milling force, therefore causing more damage to the milling bur and slowing of the milling cycle. A softer material like Enamic, on the other hand, will cause less damage to the burs and will require shorter milling time. Interestingly, no statistical difference was noted between unfired Celtra Duo and Emax.

Table 4: Mean Vickers hardness of the tested materials based on articles in the literature. Values converted from SI Units MPa and GPa to Vickers Hardness Number. Red color represents statistical difference.

	Zirconia	ZLS	Emax	Vita Enamic	BEGO
Current study results	*951.9 (94.2)*	*545.8 (54.7)*	*481.2 (28.2)*	*179.2 (17.0)*	*21.4 (2.0)*
Elsaka et al. (2016)		*665.9 (46.91)*	*555.7 (28.55)*		
Lawson et al. (2016)		*Unfired: 463.5 (26.6) Fired: 595.1 (37.6)*	*452.9 (16.2)*	*157.2 (14.0)*	
Traini et al. (2016)		*Unfired: 693.4 (50.98) Fired: 775 (71.38)*			
Mavriqi et al. (2022)		*VS:775 (71.38) CD: 724 (50.98)*	*622 (30.59)*		
Goujat et al. (2018)			*609.8 (95.85)*	*239.6*	
Elmougy et al. (2018)			*516.4 (13.36)*	*201.5 (1.224)*	
Grzebieluch et al. (2021)				273.42 (27.11)	*vertically: 25.8 (0.7) 45°: 28.16 (1.42)*
Sonmez et al. (2018)			*591.4 (10.2)*	*Before thermocycling: 234.5 (10.2) After: 193.7 (10.2)*	
Albero et al. (2015)			*594.5 (7.138)*	*173.3 (12.24)*	

Shakibafard et al. (2023)				*255.46 (3.02)*	

Traini et al. (2016) (52) also researched the effect of firing crystallization on the composition and mechanical properties of ZLS. Vickers hardness was statistically higher in the fully crystallized group. The authors emphasized the importance of thermal crystallization treatment on the optical and mechanical properties of ZLS. Their ZLS specimens were analyzed under a scanning electron microscope (SEM) (figure 10), this microstructural analysis showed a change in structure from a round nanoparticles of homogenous material (a) to a multicomponent system featuring a diffuse nano-crystal growth (b) after crystallization. This was attributed to a nucleation process following the heat treatment leading to a very fine-grained structure. It was also noted that Zirconia particles weren't identified.

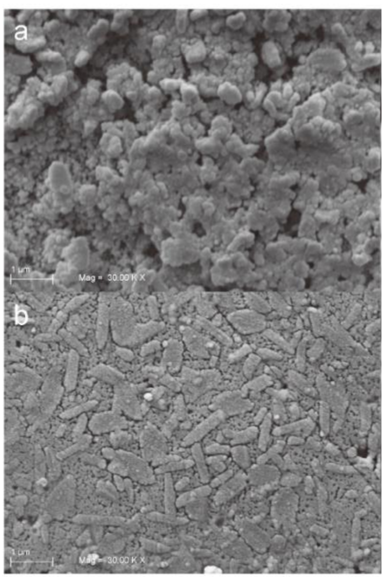

Figure 11: Scanning electron microscope of ZLS specimens before and after crystallization. SEM shows change in ZLS structure from a round nanoparticles of homogenous material (a) to a multicomponent system with a diffuse nano-crystal growth (b) after crystallization.

Mavriqi et al. (2022) (53) also conducted microstructural analysis and compared mechanical properties of two brands of ZLS (Vita Suprinity ZLSS and Celtra Duo ZLSC) with Emax. They concluded that after crystallization both ZLS materials showed higher fracture toughness and Vickers hardness than Emax. Although Vita suprinity showed higher VH values than Celtra Duo, difference between both materials wasn't significant ($p>0.05$).

Prior to heat treatment for crystallization, their microstructural analysis revealed a uniformly fine structure in all three glass ceramics with some distinctions: LS_2 displayed a prevalent presence of sub-micron platelet-shaped crystals of lithium metasilicate (Li2SiO3) and a smaller quantity of rounded nanometric crystallites of lithium orthophosphate (Li2PO4). ZLSS exhibited a nucleated and pre-crystallized microstructure, while ZLSC appeared to be in an advanced crystallized state, suitable for clinical use (Fig. 1a–c).

After the crystallization process, LS_2 demonstrated predominantly interlocking needle-shaped crystals of lithium disilicate (Li2Si2O5) embedded in a glassy matrix. Both ZLSS and ZLSC showcased rounded and road-like crystals of lithium disilicate/metasilicate, lithium monosilicate, aluminum silicate, and a glassy matrix enriched with tetragonal zirconia. In summary, ZLS materials exhibited a uniform fine crystalline structure containing rounded and rod-like crystals (Li2O-ZrO2-SiO2), particularly evident in the post-crystallization state (Fig. 1d–f)

Overall, the crystallization process of these glass materials is found to be highly dependent on temperature and results in smaller crystal size in ZLS as compared to Emax. This consequently increases the material's ability to resist permanent deformation (Hv) and contain crack propagation (ft). Celtra Duo and Vita Suprinity show statistical difference in crystal size and fracture toughness but no significant difference in VH. The authors concluded that ZLS's Hv and ft allow it to be used clinically even in posterior segments of bruxers (600-900 N masticatory force).

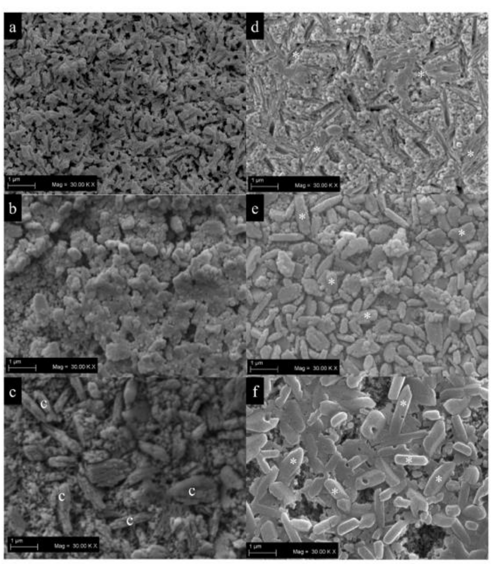

Figure 12: SEM images of glass ceramics before and after thermal treatment for crystallization (53). LS_2 IPS Emax-CAD (a), ZLS Vita Suprinity (b), and ZLS Celtra Duo (c) exhibited homogeneous material nanoparticles within the glassy matrix before treatment. Post-thermal treatment, structural changes were observed, with LS_2 displaying a significant glassy matrix, and morphological differences, such as needle-shaped crystals in LS_2 and globular/rod-like crystals in ZLS, were noticeable both before and after crystallization.

Goujat et al. (2018) (54) evaluated mechanical properties and internal fit of four CAD-CAM materials, including Vita Enamic and Emax. VH of Emax was significantly higher than all tested materials. The authors also reported that reduced hardness and modulus of elasticity lead to greater amount of material removal during grinding (31), while contrasting findings suggest that materials with lower brittleness exhibit decreased edge chipping and enhanced machinability (55–57).

Elmougy et al. (2018) compared mechanical properties of three machinable polymers (two composite and third is Vita Enamic) and Emax. It was found that the latter has significantly higher VH.

Grzebieluch et al. (2021) (58) compared mechanical properties of different printable and machinable dental composite CAD/CAM materials. Vita Enamic was found to have significantly higher Vh (the hardest) than BEGO and other composite materials. VarseoSmile Crown Plus (BEGO) was printed in two different angulations, the first vertical and the second at 45 degrees. Printing angulation wasn't found to affect VH. The material with the highest hardness in this set is Vita Enamic, attributed to its ceramic scaffold structure. It was noted that other composite materials despite having a comparable filler content, exhibited significantly lower hardness, possibly due to the absence of connections between filler particles and their smaller dimensions. A positive correlation was thus reported between filler content (by volume) and microhardness, this was consistent with findings by Ling et al. (59), Chung (60) and Mirica et al. (61). Material hardness may also correlate with abrasion, suggesting that materials with lower hardness could be more prone to wear (45). Consequently, it can be inferred that the wear of printed materials will likely progress more rapidly than that of milled materials.

Sonmez et al. (2018) (62) performed microstructural characterization and mechanical testing before and after thermocycling on five CAD-CAM materials including Emax and Vita Enamic. Ceramic-resin materials (Vita Enamic) have significantly lower Vh compared to glass-matrix ceramics (Emax). Thermocycling significantly affected mechanical properties of Enamic but not Emax. Plausible explanation is that it induced water assimilation within the resin structure, causing an expansion in the Enamic network and simplifying the frictional forces among polymer chains (27,63,64). Furthermore, there was speculation that the absorbed water could induce hydrolysis of the interfacial silane coupling agent responsible for the chemical bond between the resin matrix and the fillers.

Emax materials, in contrast, exhibited no water absorption. SEM analysis substantiated these observations: following thermocycling, Enamic images displayed degraded homogeneous structures with observable microcracks, while no changes were detected in Emax.

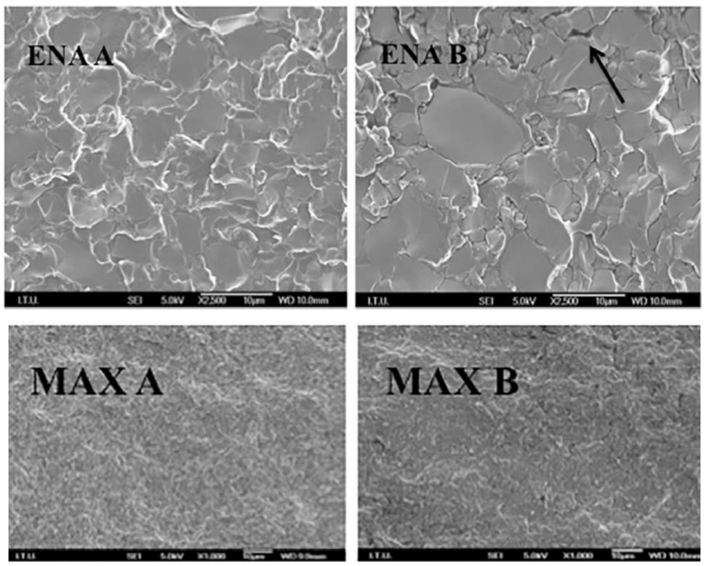

Figure 13: Effect of thermocycling on Vita Enamic (ENA) and Emax (MAX). Multiple microcracks (arrow) were noted in Vita Enamic after thermocycling (ENA B). No changes in the structure of Emax was noted after thermocycling (MAX B) (62).

Albero et al. (2015) (65) also confirmed that, Vita enamic had significantly lower Vickers hardness than Emax-and attributed that to their lower inorganic content.

Shakibafard et al. 2023 (66) in a study evaluating the effect of different teeth whitening agents and concentration on Vita Enamic noted that the material’s VH is 255.46 (3.02).

Although no studies have been conducted on Vickers hardness of BEGO, it could be concluded from this study that a low VH of 21.4 (2.0) implies a greater vulnerability to surface degradation and wear, potentially resulting in heightened surface roughness and an increased accumulation of plaque. On the other hand, a softer material causes less wear of the opposing dentition (30). Clinical application of this material can be restricted to long-term transitional or temporary crowns. It’s crucial to highlight that the assessment of a lower Vickers Hardness value should not stand alone, as it does not necessarily imply a higher wear rate; the wear rate is also influenced by the material's coefficient of friction.

Limitations

This preliminary study only evaluated Vickers hardness of the five tested materials. Evaluation of all mechanical properties as well as clinical performance of these materials is required to provide reliable conclusions and recommendations to practitioners.

Another limitation is the use of shade A1 and high translucency only. Different shades and translucencies of these materials might have different mechanical properties. Further studies on this topic need to be performed to assess the relation between shade, translucency and mechanical properties.

Moreover, the materials underwent testing in ideal settings and were kept in dry atmospheres. To enhance result precision, it's imperative to subject these materials to testing conditions that mimic the oral environment, such as thermocycling and exposure to saliva.

Conclusion

The results of this study lead to the conclusion that there is a statistically significant difference in Vickers Hardness among all the CAD-CAM materials investigated. The Vickers Hardness classification for the tested materials is as follows: Zirconia > Celtra Duo > Emax > Vita Enamic > Varseosmile Bego.

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